

On the Devitrification of Silica Glass.

By Sir WILLIAM CROOKES, O.M., For. Sec. R.S.

(Received February 15,—Read March 7, 1912.)

The use of apparatus blown and worked from melted quartz is now almost universal in chemical laboratories, especially where temperatures are required above the heat at which glass softens.

When working at fairly high temperatures, I was inconvenienced by the leakage of air through silica glass.* The apparatus (fig. 1) was in the form of a perfectly clear and transparent tube, 1 cm. diameter and 20 cm. long, with a bulb $2\frac{1}{2}$ cm. diameter blown on the end. The other end of the silica tube was drawn out for connecting with the pump and sealing. It was exhausted to a high vacuum and heated to near redness along its whole length to remove any gas that might be condensed on the walls—it was then sealed off.

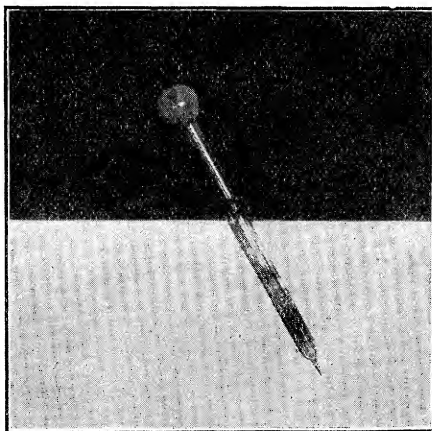


FIG. 1.

The tube was placed bulb uppermost in an electric resistance furnace in such a position that the bulb would be at the point of greatest heat, the lower part of the tube remaining comparatively cool; it was kept at a temperature of 1300° for 20 hours, at the end of which time the silica tube was removed from the furnace. The long continued high temperature

* Jaquerod and Perrot have shown that fused silica is permeable to helium and hydrogen at a low red heat ('Comptes Rendus,' Nov., 1904, vol. 139, p. 789; and Jan. 1907, vol. 144, p. 135).

caused the bulb and the upper part of the tube to devitrify, and become white and translucent like frosted glass. The sealed-off end was carefully opened, and it was apparent that the inrush of air was by no means so strong as it would have been had the vacuum been as perfect as it was when the tube was sealed up.

This looked as if there had been a considerable amount of leakage through the devitrified bulb, and I tried a test experiment. The tube was again attached to the Sprengel pump, and exhausted to as high a point as possible. During the progress of exhaustion, when the pump was rattling with the characteristic sound of a high vacuum, a large and powerful Bunsen flame was used to heat the bulb. Not the least difference in the sound could be distinguished. When the vacuum was at its highest the tube was sealed off; it was put into the electric furnace, and kept at a temperature of 1300° for 11 hours. After cooling, the end of the tube was broken off under mercury. The mercury rose, but did not fill the bulb. The amount that entered was measured, and found to be 17.75 c.c. Afterwards the tube and bulb were completely filled with mercury, the whole again measured, and the capacity of the tube and bulb was found to be 19.25 c.c., showing that 1.5 c.c. of gas, or 7.79 per cent. of the tube's capacity, had leaked through the devitrified silica in 11 hours at 1300° .

To ascertain if air would leak through the devitrified silica at the ordinary temperature a facsimile of tube and bulb was made in glass, and the two tubes were simultaneously exhausted on the pump. They were both heated, allowed to cool, and sealed off at the same time. The silica and glass tubes were put in the balance case and kept there for some time. When they were both at uniform temperature the silica tube was weighed. The tube and weights not being moved in the meantime, weighings were taken hourly, the balance being untouched during the intervals. In 18 hours the weight increased 0.048 grain.

After the silica and glass tubes had been at rest for some days, they were opened simultaneously under mercury. The glass tube filled at once, only a microscopic bubble of air remained at the top. The silica tube, on the contrary, only partially filled, and, on measuring the mercury that entered, it amounted to 10.15 c.c., the capacity of the tube being 19 c.c. Therefore, in a few days, air to the amount of 46.58 per cent. of the total capacity of the apparatus had leaked in.

A micro-photograph was taken of the surface of the devitrified silica bulb (fig. 2). It showed a surface cracked all over into the appearance of cells, and, on closer examination, many of the cells showed decided hexagonal outline.

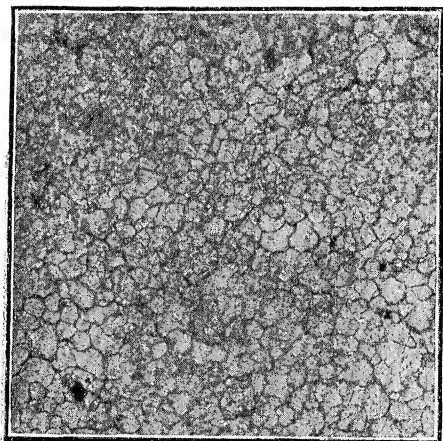


FIG. 2.

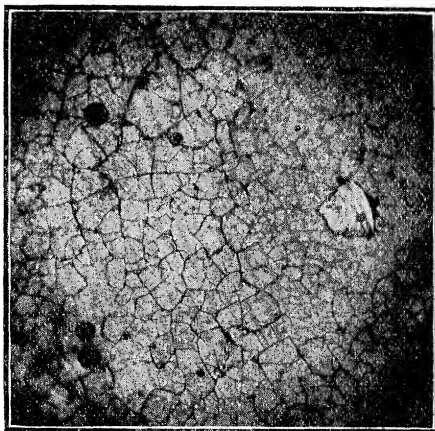


FIG. 3.

I observed a similar appearance a few years ago, when a silica dish, originally clear and transparent as glass, was used for evaporating down about 100 mgrm. of pure radium bromide. Patches appeared on the bottom having a dull, roughened appearance, and, on examination under the microscope, the appearance was very similar to the surface of the devitrified silica bulb just described (fig. 3). The appearances are so alike that it is legitimate to assume that the same cause had been at work, and that devitrification of the surface is produced both by exposure to a very high and long continued temperature and to the contact with a radium salt at a temperature of boiling water. I have not seen this effect on the surface of glass or silica bottles in which radium salts have been kept in the cold for some years.



FIG. 1.

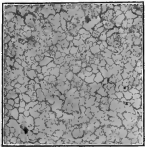


FIG. 2

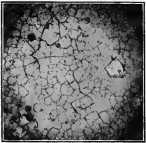


FIG. 3.